

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/691,052
Applicant : Milliren, Charles M.
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Title : VISCOELASTIC FOAM LAYER AND COMPOSITION
Examiner : John M. Cooney
Docket No. : 36211
Customer No. : 000116

DECLARATION OF CHARLES M. MILLIREN, Ph.D.

Charles M. Milliren, Ph.D., having knowledge of the facts set forth herein, declares as follows:

1. I am the named inventor of the present patent application.
2. I presently reside at 12589 Harold Drive, Chesterland, OH 44026.
3. I received a Bachelor of Science in Chemistry from Ursinus College, Collegeville, PA in 1969. I received a Ph.D. in Organic Chemistry from the University of North Carolina at Chapel Hill, North Carolina in 1975.
4. Following my formal education, I was employed by Mobay Chemical Corporation/Miles Inc., Pittsburgh, Pennsylvania, as a research scientist from 1976 to 1991. Subsequently I worked for Bayer Corporation USA in Pittsburgh, Pennsylvania, also as a research scientist, from 1991 to 2001. From 2002 till the present I have been employed by Coit Road Incubator, Cleveland Ohio and engaged primarily in the research, development and testing of foams for energy absorption applications.
5. I am an expert in foam chemistry, particularly polyurethane foam chemistry and morphology, with over 29 years of professional research and development experience. I have authored or co-authored several publications in the

field of polyurethane foam chemistry and morphology, a partial list of which is attached hereto as Attachment A.

6. I also am an inventor or co-inventor of numerous U.S. patents relating to polyurethane foam chemistry and morphology, which are listed in Attachment B.

7. Viscoelastic foams are a class of foams that exhibit aspects of both viscous and elastic character. Foams in this class are deformable or deflectable based on either a dynamic impact event or a quasi-static compression event, but there is no permanent deformation once the external stress or force causing the deflection is removed. However, the rate of recovery of viscoelastic foams is not instantaneous and includes a time dependent component based on the foam's unique morphology. In simpler terms, there is a prolonged or extended process of recovery after the external compression or impact load has been removed, the duration of which is based on the foam's morphology, which is determined based on its unique composition. To better understand viscoelastic foams, a brief discussion of purely elastic and purely viscous foams is provided below.

8. For a purely elastic foam, all the energy of loading or compression (i.e. the energy required to compress or deflect the foam) is stored in its compressed or deflected state, and is returned by the foam once the load has been removed. Another way to think of it is that the foam essentially pushes back with the same force (equal and opposite) that is acting against and tending to compress it, and it continues to push back, even under static conditions, with the same force until the load is removed. The displacement of an elastic foam exhibits an immediate (non-time dependent) response in phase with the load or strain.

9. Conversely, for a purely viscous foam, no energy is returned after the load tending to compress the sample is removed. More simply, a purely viscous foam does not "push back" against the loading stress except to the extent based on the foam's "rigidity" (described below). In other words, a viscous foam does not exert any rebound force against compression under static conditions (i.e., once the object supplying the compression force has come to a stop in contact with the compressed foam). Instead,

all the energy imparted to the foam to compress it is converted into internal energy or heat based on internal frictional losses from the viscosity of the foam material.

10. All foams that are neither purely elastic nor purely viscous are classified as "viscoelastic" foams. In a viscoelastic foam, some of the loading energy (stress) from an external impact is returned by the foam following removal of the external load, and the remainder is dissipated through conversion to internal energy or heat from internal frictional losses based on the material's viscosity. The proportion of the loading energy (stress) that is lost in this fashion, and not returned by the foam, is called hysteresis. A typical hysteresis loss for a viscoelastic foam is greater than 60%, and can be as high as 80% or more, of the loading energy. The high hysteretic characteristic of viscoelastic foams makes them desirable for applications requiring low rebound rate and high-energy absorption. From the foregoing, it should be evident that viscoelasticity refers to the manner and mechanism through which a foam stores and/or dissipates the energy required for compression.

11. Another material property particularly germane to the characterization of foams is rigidity. The rigidity of a foam is a measure of the foam's static hardness, sometimes called its "durometer" or modulus. More specifically, rigidity refers to the qualitative hardness or resistance to deflection of a material, such as a foam, based on an external load. The rigidity of foams can be understood through analogy to the rigidity of objects of more common experience. For example, a rubber ball (such as a racquetball) is less rigid than a baseball, and a baseball is less rigid than a bowling ball. A foam that is truly rigid cannot be deflected without destruction of the foam. In practice, very few if any foams are truly 100% rigid, as all (or most) foams can be deflected to some finite degree based on a quantifiable modulus of elasticity or "Young's modulus." But for practical purposes, a foam that will break rather than yield (bend or deflect but remain structurally intact) to any significant degree on being loaded beyond a threshold value is considered a rigid foam. A good example of a rigid foam is expanded polystyrene commonly found in bicycle helmets. Expanded polystyrene is very rigid; i.e. it is not susceptible to being compressed or deflected to any significant degree, and if

loaded beyond a threshold value it will irreversibly, destructively crush or break rather than bend.

12. Conversely, a truly flexible (i.e. non-rigid) foam will bend or deflect readily on application of an external load. Again, in practice few (if any) foams are truly 100% flexible, as most (or all) foams will tend to resist deflection to some finite degree, at least from static inertia or the foam's elasticity. But, for practical purposes, a foam that deflects or compresses readily in response to a load is considered a flexible foam.

13. A "semi-rigid" foam is one that is neither flexible nor rigid in practical terms. Instead, a semi-rigid foam exhibits substantial resistance to deflection based on an external load, and may nondestructively yield (bend or compress) at some threshold loading value. Under normal conditions where a semi-rigid foam will encounter ordinary loads (below a threshold loading value), the foam will behave essentially as a rigid solid. But the semi-rigid foam will be nondestructively deflected or compressed in response to an external load above the threshold value. As will be understood by persons having ordinary skill in the art, the threshold value of external loading force required to cause a semi-rigid foam to deflect will be significantly greater for a high speed, dynamic impact than for a very low speed impact or a static (or quasi-static) load, due in part to the static inertia of the semi-rigid foam material. The static inertia of the foam, which by itself tends to resist deflection, coupled with the foam's morphologic resistance to deflection (i.e. its "rigidity"), compounds the foam's effective rigidity against a high speed, dynamic impact.

14. As is understood in the art, the degree of rigidity of a foam is related to the foam's viscoelasticity, but it is, nonetheless, an independent property. For example, a highly resilient ("elastic") foam will return all the energy stored in the foam that was required to deflect it once the external load has been removed. But, such a foam can be, independently, either a flexible foam, a semi-rigid foam or (rarely) a rigid foam depending on its resistance to being deflected. Conversely, a low-resilient or "viscoelastic" foam will recover in a time-delayed manner and return only a portion of the energy required to deflect it based on the hysteresis function of the foam. But, such

a foam also can be, independently, either flexible, semi-rigid or rigid depending on its resistance to being deflected.

15. The specific chemical components that go into a polyurethane foam are outcome-determinative for the physical and behavioral characteristics just described for the resulting foams. In addition to polyol(s), polyurethane foam formulations may include blowing agent(s), catalyst(s), chain extender(s), crosslinker(s), pigment(s), surface active agent(s), filler(s), flame retardant(s), etc., and polyisocyanate(s). Each component can affect the characteristics of the final foam in an unpredictable way based on its effect on the foam's morphology and physico-chemical structure, which cannot be specified *a priori* and often defies explicit characterization even after the foam is made. In general, the polyols used in polyurethane foams can be polyether or polyester types. For polyether foams the initiator molecule and the alkyleneoxides used for chain extension and tipping are extremely important in determining the solubility, compatibility, reactivity, surface tension, stability and function of the reacting polyol. The alkyleneoxides make an important contribution toward determining the physical characteristics of the resulting polyurethane foam, i.e., the foam's morphology, which is largely determined by the molecular weight, structure and reactivity of the polyol(s) in addition to other factors. The morphology determines the type and amount of important physical interactions in the resulting foam. These interactions include the extent of hydrogen bonding in the foam and the hard block/soft block distribution in the foam, which contribute to the foam's rigidity. Importantly, the glass transition temperature, the foam's rigidity and its viscoelasticity all are determined by the foam's morphology in a largely unpredictable way.

16. It is safe to say that if the components of two polyurethane foam compositions are not precisely identical, then the resulting foams will have different physical properties. Two foams may be "semi-rigid" (semi-flexible) polyurethanes but differ in their viscoelasticity (resilience), density, tensile strength, compression set, impact character, tear strength, compression hardness, etc. Similarly, two foams may be "flexible" polyurethanes and differ from one another in similar fashion. Conversely, viscoelastic foams, which have in common the characteristic that they exhibit slow

recovery and thus high hysteresis during a compression cycle, can be flexible, semi-rigid (semi-flexible) or, although rarely, rigid as is understood by those with experience in the field.

17. Based on the foregoing, the patent examiner's characterization of "semi-rigid" as not lending to the claims a patentable limitation was incorrect because in the foam art this term describes the physical structure of the foam. In summary, a "semi-rigid" foam resists deflection from an external load to a significant degree comparable to rigid foams (e.g. EPS), but nevertheless can be nondestructively deflected and will recover following such deflection commensurate with the foam's relative elasticity (unlike a rigid foam which would be destructively deflected or crushed and could not recover). The foregoing is a qualitative physical property of the foam based on its chemical make-up and the resulting structure and morphology. This property cannot otherwise effectively be described, for example based on the precise physico-chemical structure that produces the observed behavior, because the precise structure of a foam defies explicit characterization through conventional techniques. Foams are extremely complex materials characterized by a very complex cross-linking structure, cellular structure, solid porosity, and other structure-contributing factors that cannot be readily quantified or observed. Thus, the physical structure of foams typically is classified using terms describing their behavioral characteristics as described herein, and "semi-rigid" is one of those terms. "Semi-rigid" is a structural limitation because it describes by necessary implication otherwise uncharacterizable aspects of physical structure which are responsible for imparting the "semi-rigid" properties to the foam as previously described.

18. Similarly, the term "viscoelastic" also is a term entitled to patentable weight for analogous reasons as above. Together, the terms "semi-rigid viscoelastic foam" as recited in the claims describe foams that are both **a)** semi-rigid in that they substantially resist deflection, and **b)** viscoelastic meaning that they exhibit hysteresis and consequent slow but substantially complete recovery following a deflection-inducing impact or load once the load has been removed.

19. Regarding the references cited by the examiner, none of these discloses or even remotely suggests a semi-rigid, viscoelastic foam.

20. The foams in Apichatachutapan are flexible foams, not semi-rigid foams, as evidenced by the data presented in the Examples of Apichatachutapan. Specifically, the foams in this reference have been tested for physical properties characteristic of flexible foams (25% and 65% IFD, sag factor) and the resulting data are typical of flexible foams; see Table 2 in Apichatachutapan. Conversely, the "semi-rigid" foams as claimed in the present application could not be tested for IFD or sag at least because they are too rigid to exhibit these properties. On the other hand, the present application presents extensive data for semi-rigid foams made according to the invention showing energy absorption characteristics at various dynamic impact speeds. Dynamic impact testing is totally unsuitable for flexible foams because such foams do not exhibit any appreciable degree of dynamic impact energy attenuation and would be instantaneously completely compressed (they would bottom out) with no energy absorption to speak of. Apichatachutapan is concerned primarily with providing a high density flexible foam that exhibits flame retardant properties without the incorporation of any separate flame retardant additive (see para. [0002] and rest of application). No mention or hint of energy absorption properties, particularly against a dynamic impact, (and hence no suggestion to prepare a semi-rigid foam) is to be found in Apichatachutapan.

21. Regarding Lutter, the foams in this reference are soft, flexible foams (see col. 4, line 45 and col. 8, line 50) which are completely different from the semi-rigid foams described in the present application. Lutter does not disclose or discuss dynamic impact properties for the foams therein, because soft, flexible viscoelastic foams are unsuited for this purpose.

22. Likewise, Falke describes only flexible polyurethane foams (col. 1, line 5) designed primarily as sound absorbers in carpet backing (col. 1 and col. 2) with a loss factor (col., line 40) greater than 0.3. Falke does not discuss or show any dynamic impact properties for the disclosed flexible foams, principally because such foams are totally unsatisfactory for dynamic impact attenuation.

23. Conversely, the "semi-rigid" viscoelastic foams of the present application are directly suited for dynamic impact attenuation applications as evidenced by the substantial dynamic impact test data provided in the specification. There would be no reason to make, or even to expect one could make, a "semi-rigid" foam falling within the scope of the present claims based on the teachings of any of the cited references, in particular because all of them contemplate flexible foams and none of them even hints at a semi-rigid foam or at any application, such as dynamic impact energy attenuation, that calls for a semi-rigid foam.

24. The present invention is based on the extremely surprising result that the combination of a substantial amount of an amine-based polyether polyol, or a mixture of such polyols, that is/are propylene oxide extended, together with at least one other filled and/or unfilled polyether polyol and an appropriate amount of isocyanate as well as with other component(s) as claimed, produces a semi-rigid viscoelastic foam that has proven very effective to attenuate impact force across a very wide range of dynamic impact speeds; e.g. ranging from 2-6 meters per second, or broader. This result was particularly surprising because amine-based polyols are usually considered to be rigid foam precursors, and it was quite surprising that the use of such polyols, in relatively significant amounts with other polyols, would produce such effective semi-rigid foams that are so well suited to dynamic impact energy attenuation applications, yet still recover following an impact. Even more surprising was the breadth of impact speeds over which impact force could be effectively attenuated using these foams compared to a conventional rigid foam such as EPS.


25. For example, Example 1 describes eight different foams all made according to compositions of propylene oxide extended amine-based polyols with filled and unfilled (trifunctional, glycerin based) polyols as claimed. Table 3 of that Example shows that all eight of these foams performed comparably to conventional expanded polystyrene (EPS), which is the conventional rigid foam used in bicycle helmets to attenuate high speed impacts, for an impact velocity of nominally 6.23 meters/second. In fact, six of the eight foam formulations tested actually exhibited lower peak g-values (breakthrough acceleration values) than EPS. This was a very highly surprising and

unexpected result for a semi-rigid viscoelastic foam that substantially recovered following each impact.

26. Example 2 and Figs. 1-12 demonstrate further the ability of an exemplary semi-rigid viscoelastic foam according to the invention, Foam No. 2 from table 2, to adequately and effectively attenuate dynamic impacts of low (2 meters/second) to high (6 meters per second and beyond) compared to conventional EPS. As the data demonstrate, EPS is far less effective than the foams of the invention to attenuate low speed impacts because of its substantial rigidity. In addition, EPS, being a rigid foam, is irreversibly and destructively crushed on impact of sufficient force and thus did not recover and could not be reused. Conversely, the foams according to the invention, being semi-rigid and viscoelastic, were effective to attenuate a wide range of impact forces and did recover substantially, permitting their reuse for multiple impacts.

27. Nothing in the prior art references suggests the combination of an amine-based polyether polyol(s), which are very reactive and typically are used for rigid foams, with the filled and/or unfilled polyols as claimed to produce a semi-rigid viscoelastic foam. Furthermore, nothing in the prior art suggests the amine-based polyol(s) should be propylene oxide extended as in the foam formulations described and claimed, and the combination of these components to produce the semi-rigid viscoelastic foam having the properties described hereinabove truly was a very surprising and unexpected result that could not have been predicted *a priori* based on selection of polyols and/or other reagents for the foam compositions. Certainly, there would have been no expectation of success, starting from any of the cited references, to produce a semi-rigid viscoelastic foam as claimed, based significantly on propylene oxide extended amine-based polyols, at least because all those references are directed to flexible foams and no reason is provided or evident in any of them why or how amine-based polyols can be used to provide an effective semi-rigid viscoelastic foam.

28. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.



Charles M. Milliren, Ph.D.

8/17/05
Date

**ATTACHMENT A:
List of Relevant Publications**

1. 33RD Polyurethanes Technical/Marketing Conference, Sept. 30-Oct. 3, 1990, pp 28-31, A New MDI Foam Technology for Seating and Headrest Applications.
2. *Plastics Engineering*, Jan. 1991, page 23, "A New Non-CFC, MDI Based Flexible Foam Technology."
3. Polyurethanes Expo 99, Sept. 12-15, 1999, pp. 151-161, "Low Compression Set – High Elongation: A Paradox?"
4. Paper by Orbseal LLC and Bayer Corporation, Oct. 1, 2001, New PU Cavity Filling Foam for Sound Abatement in Car Body Shells.

ATTACHMENT B:
List of U.S. Patents of which I am Inventor or Co-Inventor

1. 4,876,292 - Isocyanate reactive mixture and the use thereof in the manufacture of flexible polyurethane foams
2. 5,028,637 - Isocyanate reactive mixture and the use thereof in the manufacture of flexible polyurethane foams
3. 5,252,624 - System for the production of toluene diisocyanate based flexible foams and the flexible foams produced therefrom
4. 5,405,886 - System for the production of toluene diisocyanate based flexible foams and the flexible foams produced therefrom 5,415,802 Water Blown, Energy Absorbing Foams
5. 5,417,880 - Water blown, energy absorbing foams
6. 5,449,700 - Water blown, energy absorbing foams
7. 5,457,137 - Water blown, energy absorbing foams
8. 5,874,485 - Flexible foams and flexible molded foams based on allophanate-modified diphenylmethane diisocyanates and processes for the production of these foams
9. 6,235,138 - Polyurethane foam/PVC laminate for automotive instrument panels